

REPORT DOCUMENTATION PAGE

AFRL-SR-AR-TR-05-

The public reporting burden for this collection of information is estimated to average 1 hour per response, including gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments, information, including suggestions for reducing the burden, to Department of Defense, Washington Headquarters Serv 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302. Respondents should be aware that notwithstanding any penalty for failing to comply with a collection of information if it does not display a currently valid OMB control number. PLEASE DO NOT RETURN YOUR FORM TO THE ABOVE ADDRESS.

es,
n of
88),
any

0512

1. REPORT DATE (DD-MM-YYYY) 06-09-2005		2. REPORT TYPE Final Report		3. DATES COVERED (From - To) 05/15/00 - 05/14/05	
4. TITLE AND SUBTITLE Magnetic Properties of Nanocrystals		5a. CONTRACT NUMBER		5b. GRANT NUMBER F49620-00-1-0315	
		5c. PROGRAM ELEMENT NUMBER		5d. PROJECT NUMBER	
				5e. TASK NUMBER	
				5f. WORK UNIT NUMBER	
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Leland Stanford University Office of Sponsored Research 651 Serra Street, Room 260 Stanford, CA 94305-4125				8. PERFORMING ORGANIZATION REPORT NUMBER SPO #21985	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) Air Force Office of Scientific Research 875 N. Randolph St. Ste 325, Room 3112 Arlington, VA 22203-1768 NE				10. SPONSOR/MONITOR'S ACRONYM(S) AFOSR	
				11. SPONSOR/MONITOR'S REPORT NUMBER(S)	
12. DISTRIBUTION/AVAILABILITY STATEMENT Approved for public release; distribution is unlimited.					
13. SUPPLEMENTARY NOTES Some of the figures and text in this report appeared in the PhD thesis of Eric W. J. Straver.					
14. ABSTRACT This Presidential Early Career Award for Scientists and Engineers (PECASE) award was instrumental in developing new technological capabilities to study nanomagnetism and superconductivity, two enabling technologies for future Air Force systems. Three distinct technological measurement capabilities were developed: variable-temperature cantilever torque magnetometry; variable-temperature magnetic force microscopy; and large-scan-area fast-turnaround Hall probe microscopy optimized for studying coated conductors. This report focuses on the second two capabilities and in particular on their application to the problems of vortex pinning and dissipation in superconductors, which are an enabling technology for such critical Air Force needs as high-power microwave sources and superconducting generators.					
15. SUBJECT TERMS nanomagnets, magnetic nanocrystals, cantilever torque magnetometry, magnetic force microscopy, scanning Hall probe microscopy, superconductors, high-temperature superconductors, coated conductors					
16. SECURITY CLASSIFICATION OF: a. REPORT U		17. LIMITATION OF ABSTRACT		18. NUMBER OF PAGES 10	19a. NAME OF RESPONSIBLE PERSON Laraine Lietz-Lucas
b. ABSTRACT U		c. THIS PAGE U		19b. TELEPHONE NUMBER (Include area code) 650-736-2045	

Magnetic Properties of Nanocrystals

A Presidential Early Career Award for Scientists and Engineers (PECASE)
For the period May 15, 2000 to May 14, 2005

Principal Investigator:

Kathryn A. Moler

Geballe Laboratory for Advanced Materials

Stanford University

Stanford, CA 94305

November, 2005

Index

Abstract	1
Cantilever Torque Magnetometry of Nanomagnets	2
Motivation and Decision to Develop Tools to Study Vortex Pinning	3
Development and Construction of a Variable-Temperature Magnetic Force Microscope (MFM).....	4
Applications of the MFM to Study Vortex Pinning in Superconductors.....	5
Development and Construction of a Large-Area Scanning Hall Probe Microscope Optimized for Coated Conductors	7
Development of Metal-Coated Carbon Nanotube Tips for High-Resolution Magnetic Force Microscopy	9
Personnel Supported	10
Publications.....	10
New Discoveries, Inventions, or Patent Disclosures	10
Honors/Awards	10

Abstract

This Presidential Early Career Award for Scientists and Engineers (PECASE) award was instrumental in developing new technological capabilities to study nanomagnetism and superconductivity, two enabling technologies for future Air Force systems. Three distinct technological measurement capabilities were developed: variable-temperature cantilever torque magnetometry; variable-temperature magnetic force microscopy; and large-scan-area fast-turnaround Hall probe microscopy optimized for studying coated conductors. This report focuses on the second two capabilities and in particular on their application to the problems of vortex pinning and dissipation in superconductors, which are an enabling technology for such critical Air Force needs as high-power microwave sources and superconducting generators.

Cantilever Torque Magnetometry of Nanomagnets

Nanomagnets are of great interest because of their potential applications in such areas as ultra-high density information storage media, spin electronics, and magnetic sensors. Chemically synthesized cobalt nanocrystals with monodispersed (uniform) diameters on the order of 10 nm offer a potentially excellent material for such applications (figure 1).

Fig. 1. SEM Image of cobalt nanomagnets, supplied suspended in solution by the Bawendi group at MIT and placed on a substrate by AFOSR-supported student Eric W.J. Straver. The diameter of each nanomagnet in this image is 9 nm.

We characterized their magnetic properties by measuring the low temperature hysteresis loops of small numbers (~1000's) of Co nanocrystals using the technique of cantilever magnetometry, with a moment sensitivity of $10^6 \mu_B$. We were able to measure the magnetic response with sufficient sensitivity to encourage us to pursue our ultimate goal of single-nanomagnet magnetometry. However, the particles were not nearly as ferromagnetic as we had initially expected, probably owing to oxidation. After numerous runs on various samples provided by collaborators making 10-nm-scale nanomagnets, we concluded that the materials were not yet ready for our advanced characterization techniques. In the meantime, we had become very interested in the problems of vortex pinning in superconductors, so after consultation with our Program Officer, Harold Weinstock, we decided to use the career-building flexibility of the PECASE funds to develop new ways to characterize vortex pinning.

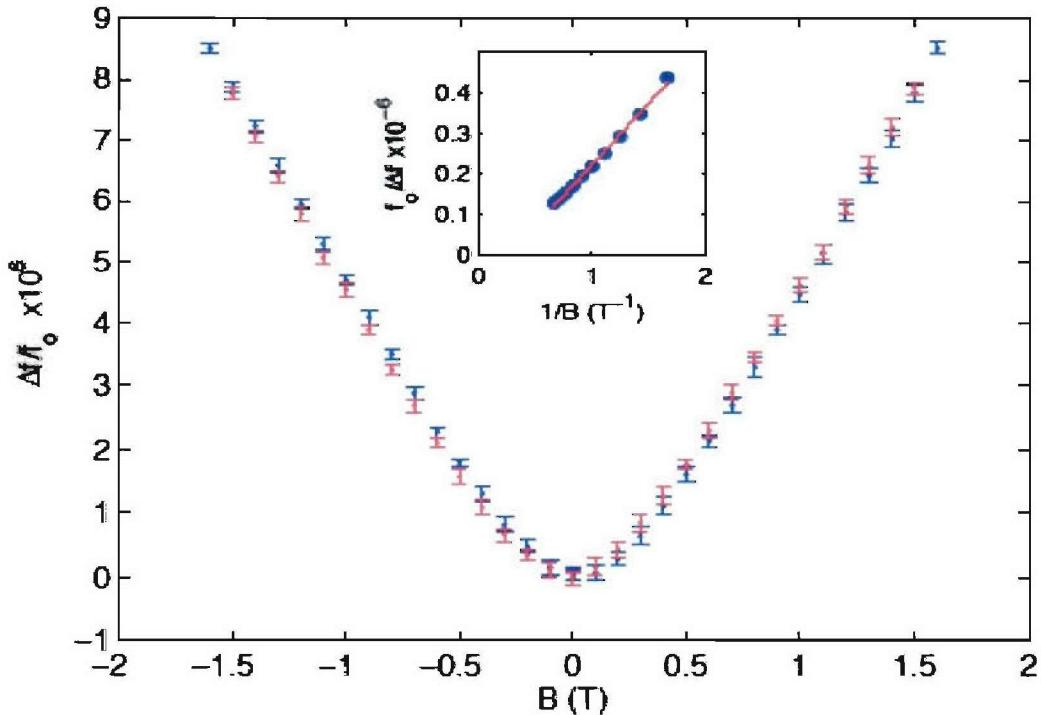


Fig. 2. Frequency shift of a cantilever with a dilute sprinkling of nanomagnets as a function of applied field at 8 Kelvin, showing a dominantly paramagnetic response with some ferromagnetic hysteresis.

Motivation and Decision to Develop Tools to Study Vortex Pinning

The relevance of high-temperature superconductors (HTS) for the Air Force lies in their enormous potential, not only for power grids, but also for key Air Force technologies such as high-power microwave sources and high-energy magnet technology. Such technology could lead to vastly improved power components such as generators, including extremely compact generators for directed energy applications, and efficient motors for electric ship propulsion. HTS are complicated materials whose utility is limited by several factors, most fundamentally by the motion of quantum whirlpools of electrons, called vortices. Vortex motion is induced by current flow and causes dissipation, thereby destroying the utility of the superconductor. Understanding the microscopic mechanisms of vortex motion and vortex pinning may enable improved current-carrying capability of HTS and achievement of Air Force objectives.

The flexibility of a PECASE award gave me the ability to construct two tools to understand the dynamics of individual vortices. The first tool is a variable-temperature Magnetic Force Microscope (MFM), which is the highest-spatial-resolution magnetic imaging tool that can be used at the surface of a sample without special sample preparation. (MFM construction was supplemented by additional funding from the Packard Foundation.) The second is a large-scan-area, fast-turnaround Scanning Hall

Probe Microscope optimized for the study of HTS Coated Conductors. The SHPM is capable of single vortex resolution and represents a substantial improvement in both spatial resolution and magnetic sensitivity over MO imaging, while still allowing large scan area (1 cm) and rapid sample turnaround.

These two tools should enable us to quantify the equation of motion of a single vortex, and to understand how vortex motion – and hence dissipation – is effected by materials structure, by currents, and by other vortices.

Development and Construction of a Variable-Temperature Magnetic Force Microscope (MFM)

Magnetic Force Microscopy (MFM) and Atomic Force Microscopy (AFM) are both based on the use of a somewhat flexible micro-cantilever with a sharp, usually pyramidal, tip. The tip may be positioned close to a sample surface. A force between the surface and the tip will cause the cantilever to deflect, while a force gradient between the surface and the tip will shift the cantilever's resonant frequency. Scanning the cantilever in a raster scan allows one to map the forces (or force gradients). Coating a non-magnetic AFM tips with a magnetic material allows one to map magnetic forces as well as atomic forces (Figure 3). Magnetic forces are long-range, while atomic forces are short-range: magnetic forces dominate images taken tens of nanometers or more from the sample, while atomic forces and topography dominate images taken within nanometers of the sample surface.

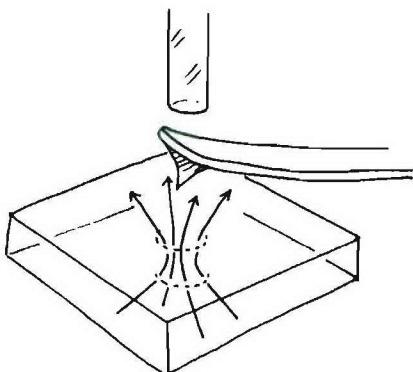


Figure 3. Sketch of Magnetic Force Microscopy showing an optical fiber, a cantilever with a sharp tip, and a sample with magnetic field lines indicating a vortex. The fiber is used to detect the cantilever detection interferometrically. Drawing by Stanford student C.W. Hicks. Not drawn to scale.

Magnetic Force Microscopy (MFM) has three advantages over other magnetic imaging techniques for the study of vortices:

1. A magnetic force microscope can also be used, in the same run, as an atomic force microscope. Therefore, it can detect surface topology effective simultaneously, and correlate defects with vortex pinning locations (first demonstrated by [Volodin 2002]).
2. A magnetic force microscope with a suitable tip can image magnetic field gradients with spatial resolution down to 20 nm, as demonstrated by this group and published in [Deng 2004].
3. The sharp magnetic tip of a magnetic force microscopy cantilever can be used to exert a force as well as to measure a force (in fact, whenever a vortex exerts a force on the cantilever, the cantilever exerts an equal and opposite force on the vortex). The MFM can be used to manipulate a vortex, and even to determine quantitatively how much

force is required to depin it, as demonstrated by this group and submitted for publication as [Straver 2004, Straver 2005].

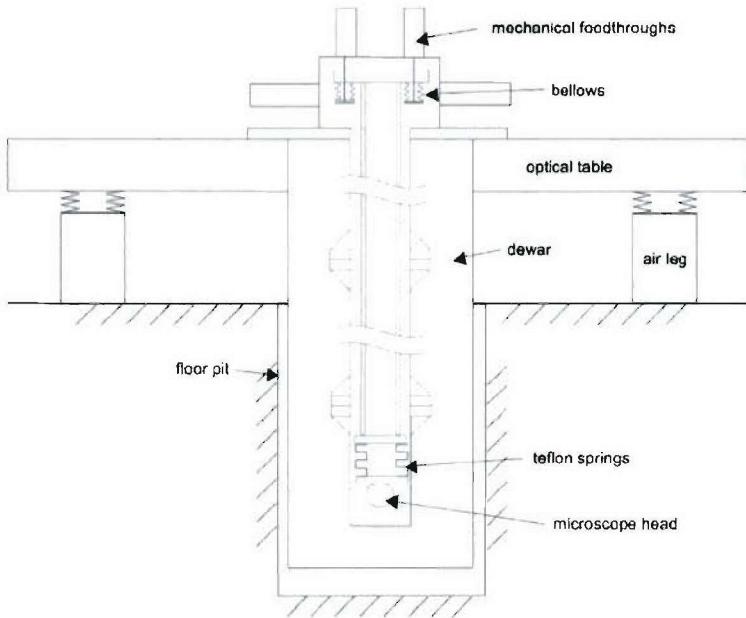


Figure 4: Schematic of the magnetic force microscope housing system, showing microscope head, dewar for cryogens, and vibration isolation.

Eric Straver (a Graduate Research Assistant supported by this grant) constructed a home-built MFM [Straver 2004] with a combination of support from AFOSR (PECASE), a Packard Foundation Fellowship, and a Canadian NSERC Fellowship. The microscope is mounted in a cryogenic dewar, which allows operation from 5 K to room temperature (Figure 4). The scan range of the microscope is over 8 microns.

Applications of the MFM to Study Vortex Pinning in Superconductors

As a first application/demonstration of the MFM, we imaged vortices and demonstrated that vortices could be directly manipulated using the MFM (Figures 5 and 6). The MFM can be used not only to manipulate vortices, but also to quantitatively measure the force required to depin them from a particular pinning site. The measured frequency shift of the cantilever is proportional to the vertical force gradient. Therefore, the frequency shift associated with a vortex, integrated as a function of cantilever-sample separation, from an infinite separation to the separation at which the vortex depins, indicates the force exerted on the vortex by the cantilever (Figure 7). A geometrical factor relates the vertical force to the horizontal force. If determined at the height where the vortex depins, this force indicates the depinning force.

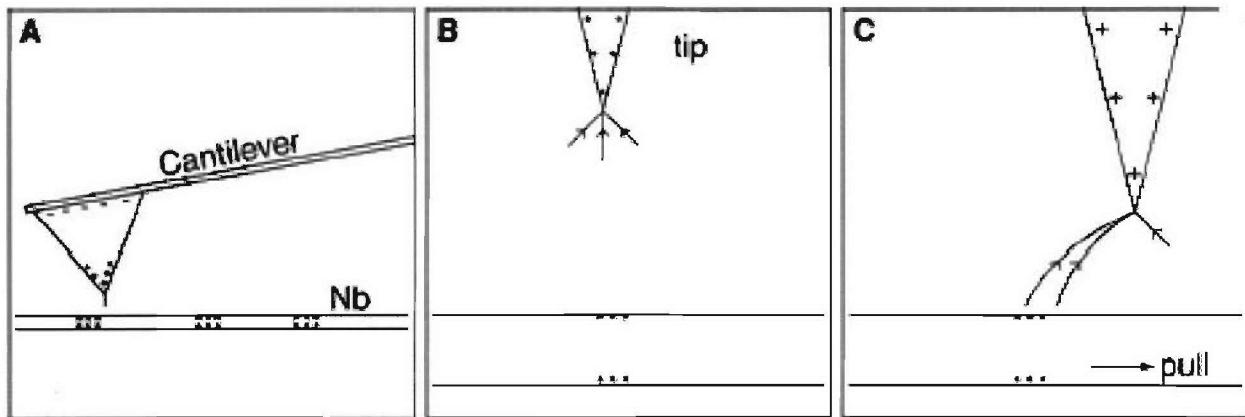


Figure 5. Sketch of the procedure for vortex manipulation. (A) Vortices are created in a superconducting film by cooling through T_c in a small magnetic field, with a polarity that generates vortices attracted to the tip. (B) At large tip-sample separations, the force on the vortex is too small to depin it, and an MFM image can be obtained. (C) When the tip is close to the sample surface, the vortex is depinned by the tip and follows it to a new location in the sample, where it finds a new pinning site.

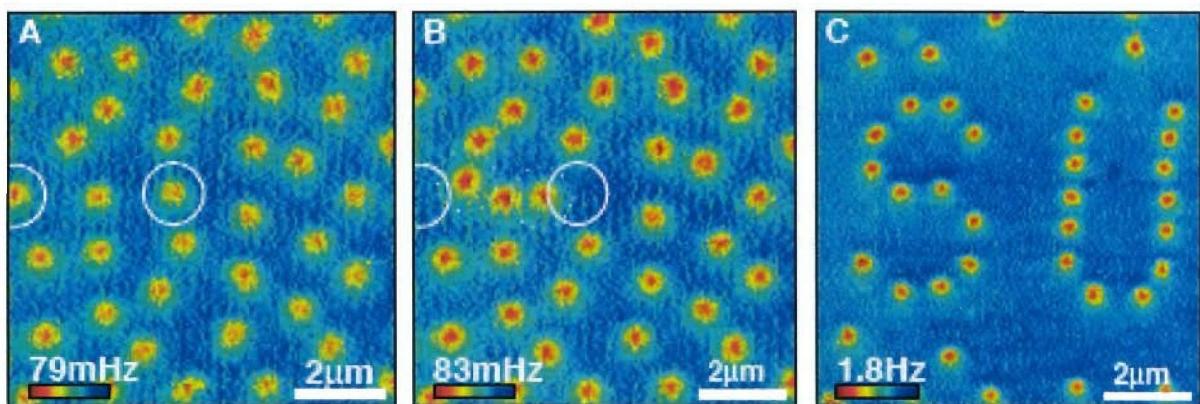


Figure 6. Vortex manipulation in Nb. (A) A configuration of vortices obtained by field cooling the niobium film to a temperature of 7.0 K. (B) Images obtained at a height of 300 nm to evaluate the success of each manipulation attempt during intermediate stages. (C) We arranged the vortices in the shape of the initials "SU", for Stanford University. This image was taken at a scan height of 120 nm and a temperature of 5.5 K, resulting in a higher quality and higher resolution image.

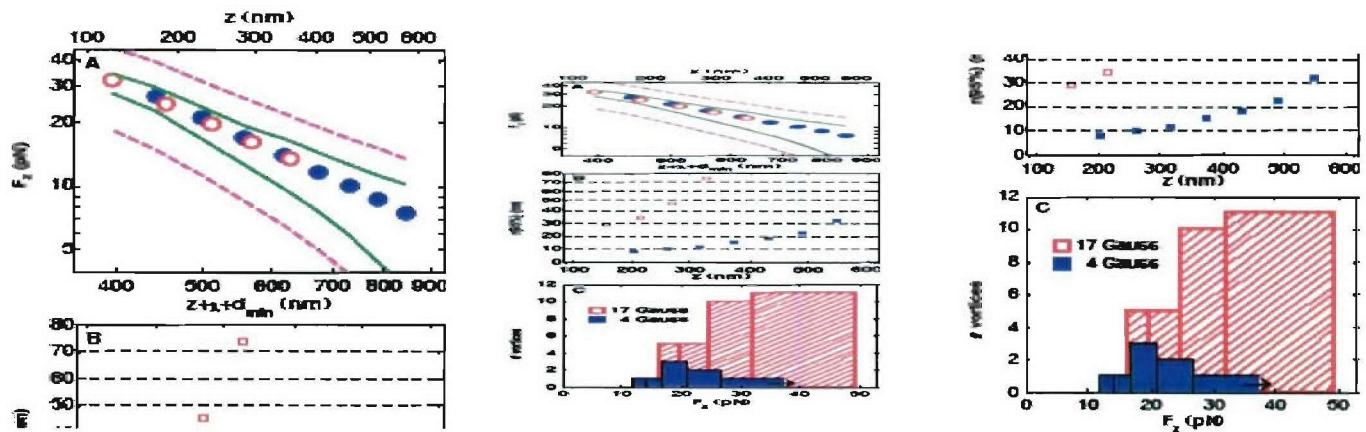


Figure 7. Quantitative depinning force measurement. (A) Vertical force vs. cantilever height. The open and closed circles show two runs: the straight and dashed lines include systematic error bars. (B) The minimum threshold for detecting vortex motion in two different runs. In the run indicated by the blue symbols, we could detect all motion on length scales of < 30 nm, or less than the coherence length, which is the physically relevant parameter. (C) Distribution of vortex depinning forces.

These results on Nb have been submitted for publication to *Science* [Straver 2005]. Ongoing work presently underway with this microscope with new AFOSR funding addresses vortex pinning in YBCO.

Development and Construction of a Large-Area Scanning Hall Probe Microscope Optimized for Coated Conductors

We constructed a scanning Hall-probe microscope that combines a 1 x 4 cm scan range with 200 nm positioning resolution by coupling stepper motors to high-resolution drivers and reducing gears (Figure 8). The instrument is uniquely suited for efficient magnetic imaging of mesoscopic devices, media, and materials, operating from 4 K to room temperature with fast turn-around time. The instrument was constructed using funds from this grant. We demonstrated its potential for studying dissipation in coated conductors—high-T_c superconducting tapes—via model systems using related MURI funding. We imaged an entire sample of YBa₂Cu₃O₇, then zoomed in to individual fluxons. The design and first results from the microscope were published in *Review of Scientific Instruments* [Dinner 2005]

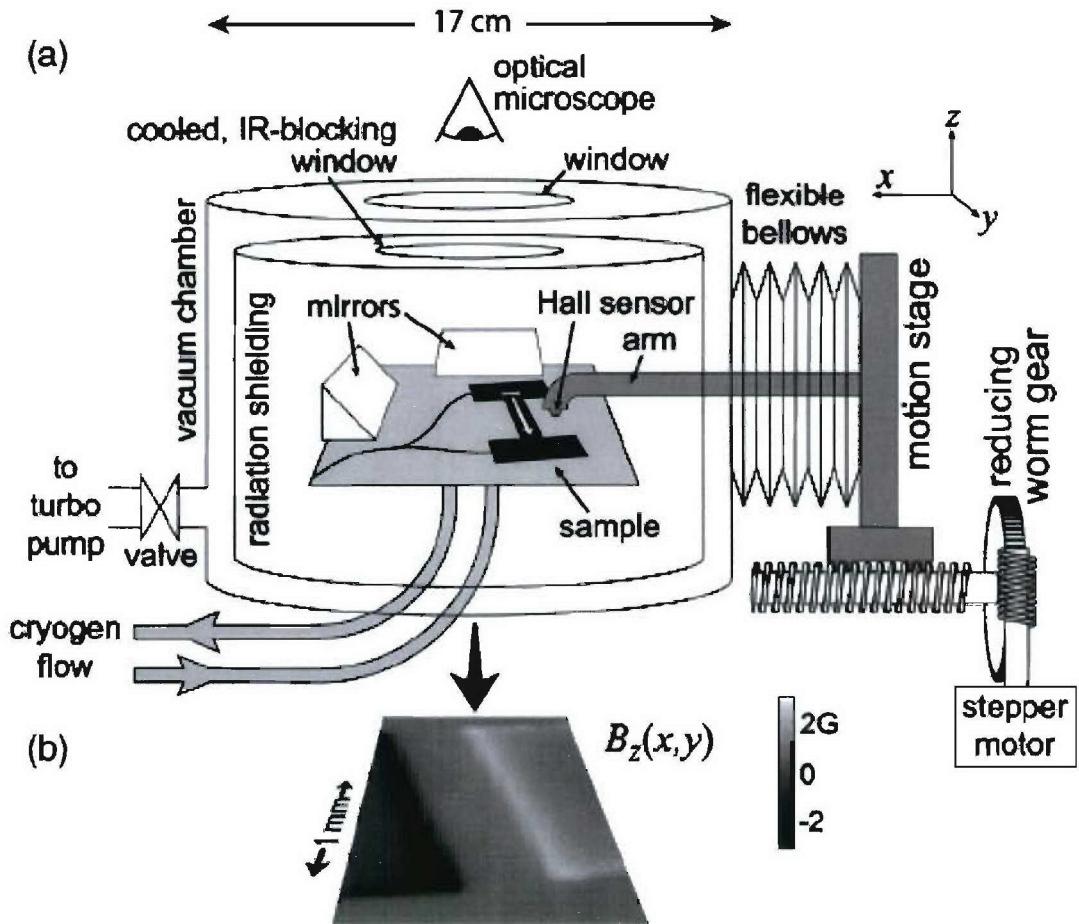


Fig.8 (a) Sketch of the large area scanning Hall-probe microscope. A flow cryostat cools the sample. A Hall sensor is rastered over the sample surface. The sensor position is controlled by an external, stepper motor-based stage. The cryostat allows optical access from above and, via mirrors, from the sides. b Magnetic image of a millimeter long YBCO strip carrying 100 mA. This image uses only a fraction of the instrument's centimeter scan range.

Development of Metal-Coated Carbon Nanotube Tips for High-Resolution Magnetic Force Microscopy

We fabricated cantilevers for magnetic force microscopy with carbon nanotube tips coated with magnetic material (Figure 9). Images of a custom hard drive demonstrated 20 nm lateral resolution, with prospects for further improvements. These results were published in *Applied Physics Letters* [Deng 2004].

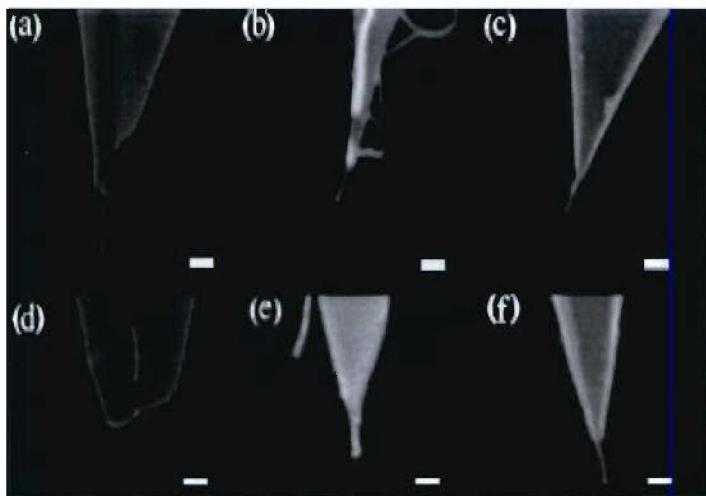


Figure 9. SEM images of six different tips with shortened, metal-coated carbon nanotube (CCNT) tips with varying coating thicknesses. Each image is on the same scale, with 100 nm scale bars shown.

FIG. 2. SEM images of six different tips with shortened, metal-coated carbon nanotube (CCNT) tips with varying coating thicknesses. Each image is on the same scale, with 100 nm scale bars shown.

Personnel Supported

Faculty: K.A. Moler

Postdoctoral Researcher: J.E. Hoffman

Graduate Student (Primary): Eric W.J. Straver

Publications

Published

[Deng 2004] Zhifeng Deng, Erhan Yenilmez, Josh Leu, J. E. Hoffman, Eric W. J. Straver, Hongjie Dai, and Kathryn A. Moler, "Metal-coated carbon nanotube tips for magnetic force microscopy," *Applied Physics Letters* **85**, 6263 (2004).

[Dinner 2005] Rafael B. Dinner, M. R. Beasley, and Kathryn A. Moler, "Cryogenic scanning Hall-probe microscope with centimeter scan range and submicron resolution," *Review of Scientific Instruments* **76**, 103702 (2005).

[Straver 2004] Eric W. J Straver, "Cantilever-Based Measurements on Nanomagnets and Superconductors," PhD Thesis, Stanford University, 2004.

Submitted, presently in referee process

[Straver 2005] E. Straver, J. E. Hoffman, D. Rugar, K.A. Moler, "Controlled Manipulation of Individual Magnetic Flux Quanta," submitted to *Science*.

New Discoveries, Inventions, or Patent Disclosures

"Metal Coated Carbon Nanotube Tip," U.S. Patent Application Serial Number 11/206,672

Honors/Awards

Eric Straver: NSERC Fellowship, 2000.

Eric Straver: PhD 2004.

K.A. Moler: Packard Fellowship, 2001.

K.A. Moler: Leigh Paige Prize Lecturer at Yale, 2004.

K.A. Moler: promoted to Associate Professor (with tenure), 2002.

J.E. Hoffman: began as Assistant Professor of Physics at Harvard, 2004.